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MATERIALS PROBLEMS IN SPACE-VEHICLE ENGINE DEVELOPMENT

ROBERT A. BOUNDY

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ENGINE DEVELOPMENT**

Robert A. Boundy



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Professional Services Section

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ABSTRACT

Materials problems associated with the design, development, and fabrication of a 6000-lb-thrust tube-bundle liquid-propellant rocket engine are discussed. Particular emphasis is given to thrust-chamber fabrication, including the development of stainless steel tubes and the brazing techniques.

Incidental aspects of fabrication are discussed, including the exit skirt, stiffener rings, wire wrapping, and chamber coating. Finally, future developments in the areas of tube fabrication and an uncooled extension nozzle are considered.

I. INTRODUCTION

There are many materials problems associated with the design, development, and fabrication of tube-bundle thrust chambers. A few of these problems will be discussed in terms of the Jet Propulsion Laboratory's 6K liquid-propellant rocket engine.

This 6000-lb-thrust regeneratively-cooled engine uses hydrazine as fuel and nitrogen tetroxide as oxidizer; a helium pressurization system is utilized. The hydrazine in particular poses many compatibility problems—for instance, several desirable brazing-alloy systems were rejected because of hydrazine incompatibility—but the hydrazine-nitrogen tetroxide system has the advantage of being storable: that is, the missile or space vehicle can be fueled in advance and fired or restarted at will.

Figure 1 illustrates the 6K propulsion system schematically. The fuel and oxidizer tanks are fabricated from 2014-T4 aluminum alloy, and the helium tank is 6AL-4V titanium alloy. The valve body and most of the components are 2024-T3 aluminum alloy and the injector is AISI 347 stainless steel except for a nickel face and splash plate. The tanks are welded. The valve and injector are brazed.

The thrust chamber is composed of a set of AISI 347 stainless steel tubes brazed together and into forward and aft manifold rings. Until recently, the manifolds were then completed by heliarc welding. The tubes have a constant wall thickness and varying cross section and contour. The chamber is wire-wrapped with Haynes alloy No. 25 and coated with a polysulfide-modified epoxy.

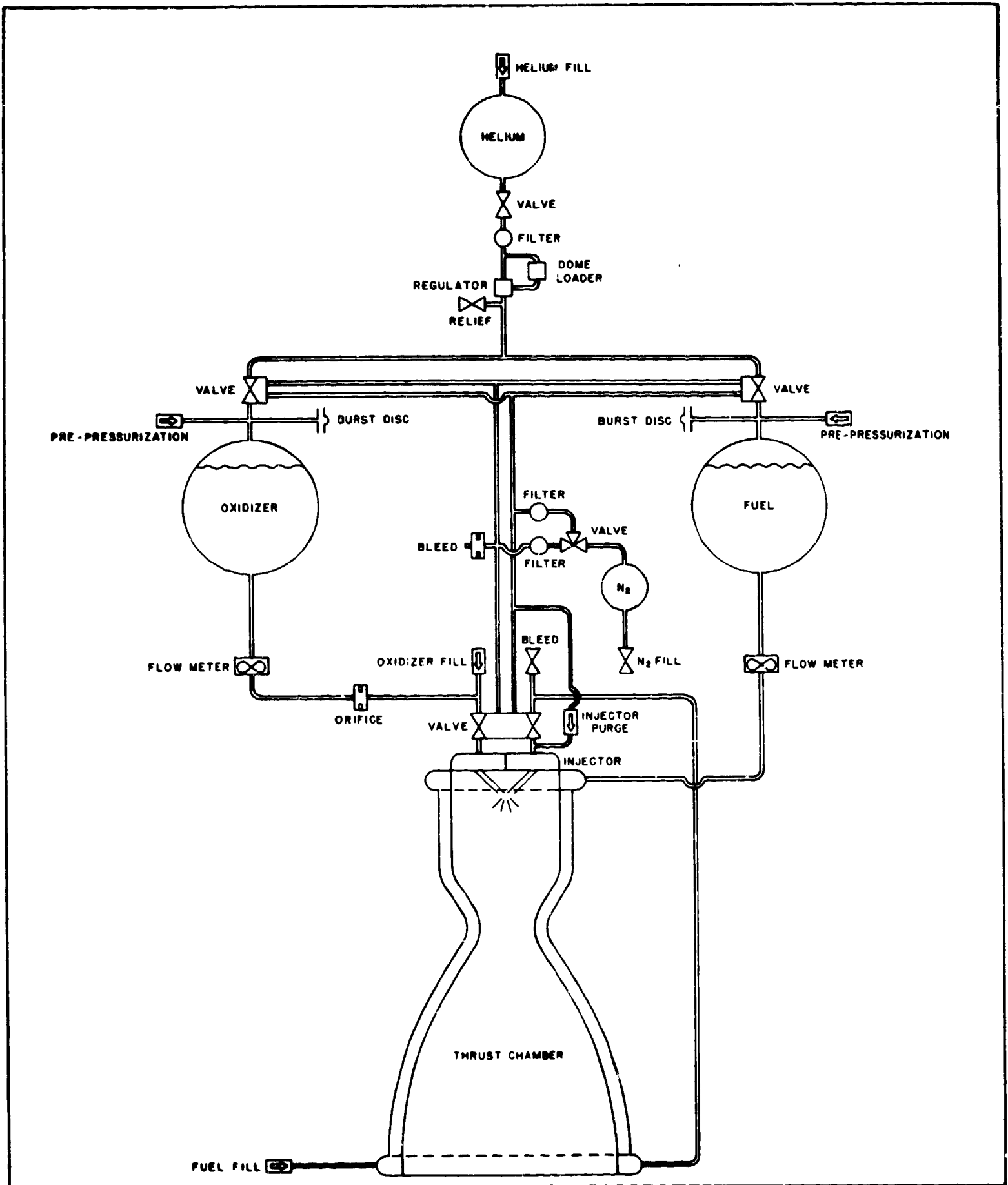


Fig. 1. 6K propulsion system.

II. THRUST-CHAMBER FABRICATION

Fabrication of the thrust chamber begins with the manufacture of the tubes, which are then flow-tested, sized, assembled on a mandrel, and brazed. The manifolds are then completed, the chamber is wire-wrapped and coated, and testing operations are performed.

A. Tube Manufacture

Manufacture of the tubes which make up the chamber wall was perhaps the most difficult problem associated with the 6K motor development.¹ Based upon preliminary design studies a stabilized austenitic stainless steel (AISI-347) was selected for prototype chamber fabrication. Both seamless and welded tubing were investigated. The welded tubing proved superior for fabrication into the complex shapes demanded by rocket thrust chambers.

The tube-wall thrust chamber is seen in cross-section in Fig. 2, which shows an assembled 20:1-expansion mockup 6K motor. A shorter 4:1-expansion motor was also designed for test purposes. Between the throat and the aft end, the tube transforms from circular to crescent and back to circular cross section. An appreciable weight saving was realized by designing the tube to have a diameter that varies with the distance from the thrust axis.

Before the actual forming begins, the straight tubes are centerless-ground externally and electrolytically ground internally, to remove the contaminated surfaces and to control wall thickness and diameter. The tubes are then fully annealed in a hydrogen atmosphere and drawn into their preformed shape. By applying tension to the tubes as they are being drawn, a constant wall thickness of 0.020 in. is maintained to ± 0.0005 in. An internal mandrel is used for some of the drawing operations.

Because of the high work-hardening rate of austenitic stainless steels, frequent process anneals are required. Until the proper balance between drawing and annealing was determined, tube cracking was a formidable problem. Internal grinding to obtain a better surface was found to ameliorate the problem. Even with elaborate

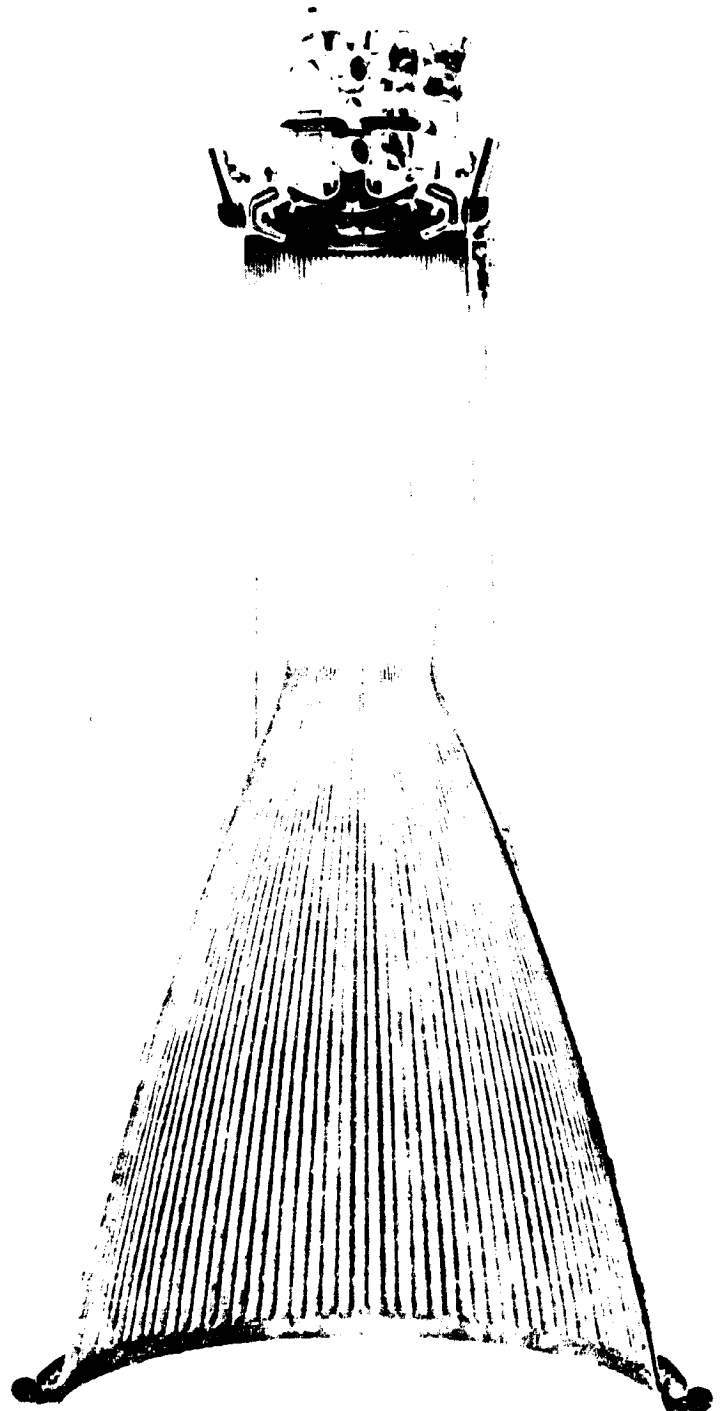


Fig. 2. Cutaway of 6K-H motor mock-up, showing tubes and manifolds.

precautions, small longitudinal cracks are occasionally noted.

In the early stages of the program, reliable non-destructive detection of internal flaws was difficult. Radio-

¹Basic tube fabrication done at Le Fiehl Manufacturing Co., Vernon, Calif., under JPL direction of L. R. Potter.

graphic inspection was useful but limited. Visual inspection of a 10% sample, sectioned and examined for pits, cracks, inclusions, etc. was considered more reliable. At present, an eddy-current inspection technique is being perfected that should prove very effective. Figure 3a shows a large pit found in a formed tube. An inclusion in a tube from the same batch can be seen in Fig. 3b.



Fig. 3. Photomicrographs of formed AISI 347 tubes showing defects: a. Pit; b. Inclusion.

The striations were caused by the severe drawing and forming operations.

B. Assembly

Most heat-resistant brazing alloys require joint clearances of 0.001-0.003 in. for optimum results. According to Chang (Ref. 1) the tensile strength and percent elongation of AISI-347 joints brazed with nickel-base alloys drops off rapidly with increased joint clearance. This subject will be explored more fully in a later section. At present it explains the need for close tolerance control on tube sizing.

Figure 4 shows a contoured and trimmed 4:1-expansion tube being sized in a book die. Experience with approximately 3000 tubes permits the following conclusions to be drawn:

1. For development work—where the tube configuration changes frequently—this method is ideal.
2. With a skillful set-up effort the average deviation at a given station can be controlled to ± 0.0005 in.
3. Considerable shimming is required to attain these dimensions.



Fig. 4. Sizing a 4:1-expansion tube in a book die.

For production the technique of hydraulically expanding the preformed tube into an enclosed die cavity seems preferable.

The sized tubes are mock-assembled on a mandrel and interference regions noted. This information, supplemented by dimensions taken at 1.5-in. increments along the tubes from a 10% sample, permit an accurate re-sizing to be made. Figure 5 illustrates an early 4:1-expansion chamber mock-assembled.

C. Brazing

The first few test motors in the 6K series were torch silver-brazed rather than furnace brazed in order to produce a prototype in the shortest possible time. Figure 6 depicts this technique. Torch brazing was a proven technique, at least for nickel tube-bundle chambers. The stainless-steel tubes, because of their lower thermal conductivity, were more difficult to braze. Concurrent with this effort, the feasibility of furnace brazing was investigated. The silver-brazing method had several disadvantages, some of which are listed below:

1. Distortions were encountered which made further fabrication difficult.

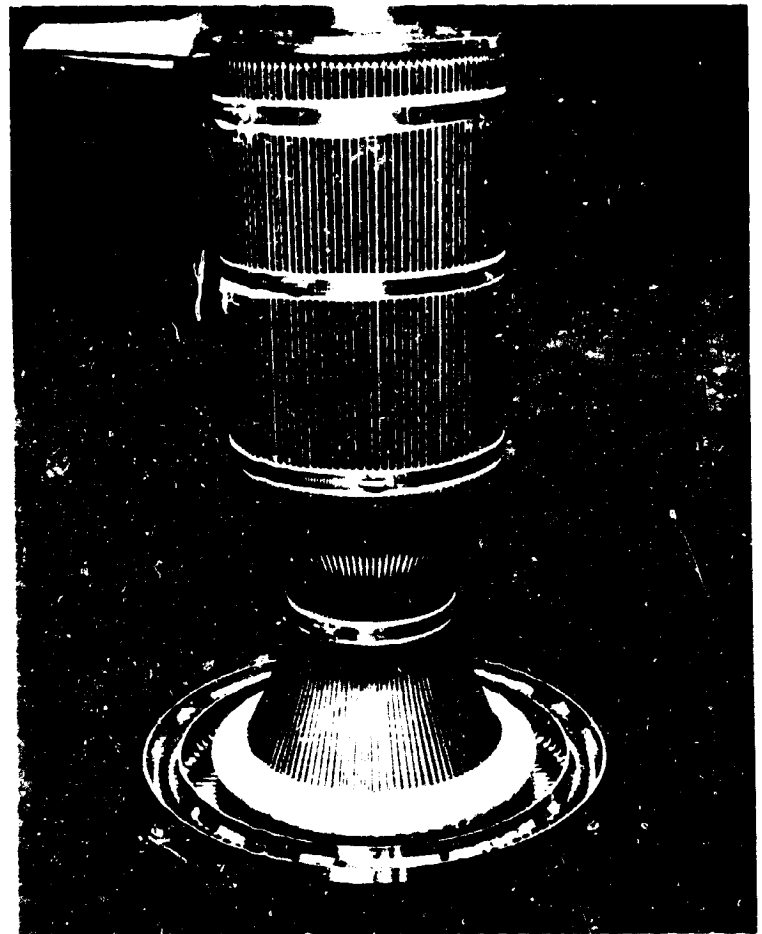


Fig. 5. Thrust chamber (6K-F motor) mock-assembled.

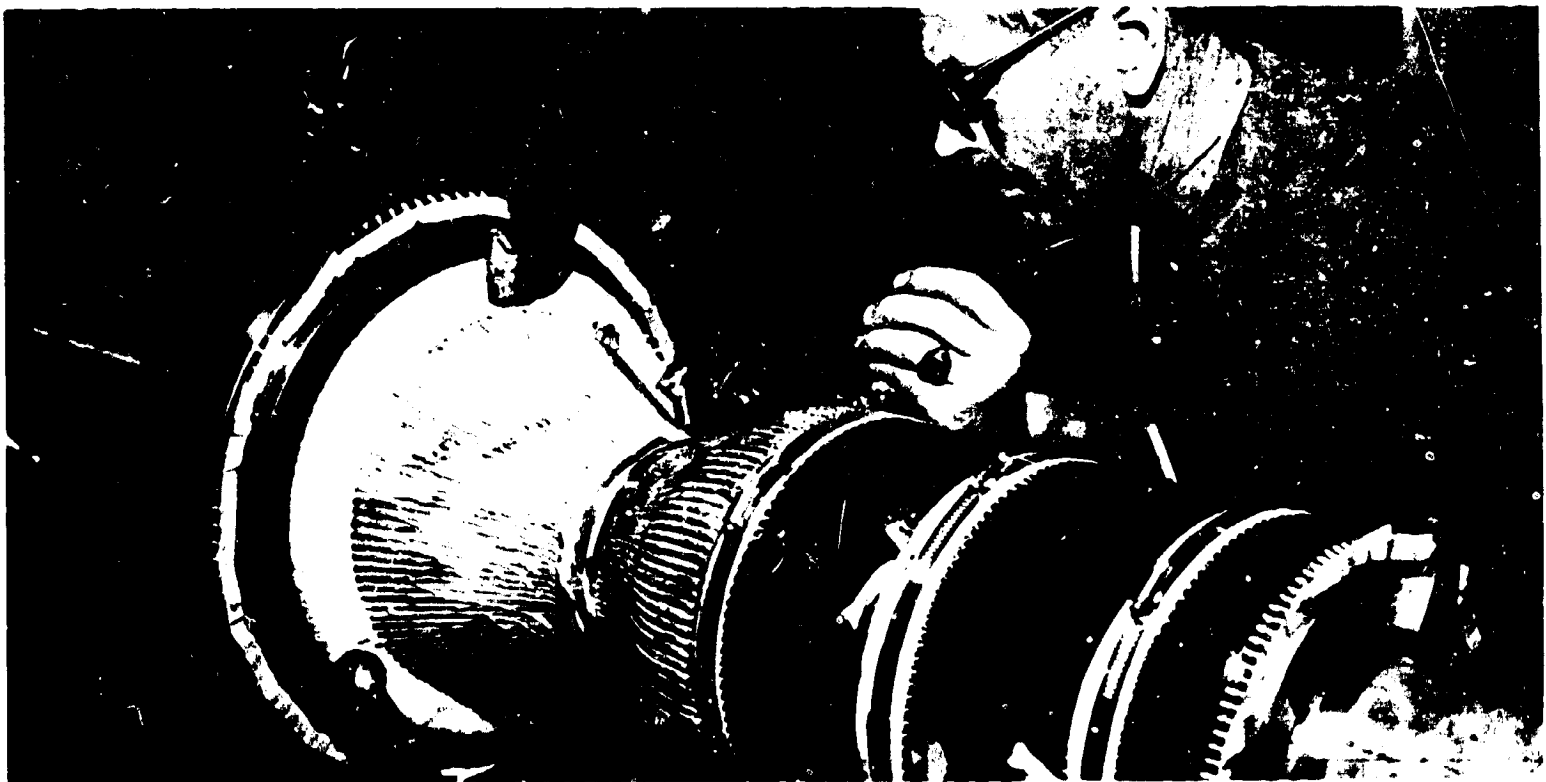


Fig. 6. Torch silver-brazing chamber (6K-A motor).

2. A compatibility problem with hydrazine necessitated welding the tube bundle to the forward and aft manifold ring.
3. It was necessary to pickle the inside of the tubes and manifold after brazing and welding, which led in some cases to pitting.
4. The recombined combustion products, if not neutralized and/or flushed out immediately after firing, tended to attack the brazed joints.

However, as a compensatory advantage, it was noted that the combustion gases formed a self-limiting coating on the silver-brazed joints, increasing the service life.

1. **Furnace brazing.** Furnace brazing, while no panacea, eliminates the four basic problems listed above. It should be remembered that this applies specifically to the 6K system; it may not hold for other systems. In furnace brazing, the tubes and related components are assembled on a mandrel, sealed into a retort or muffle purged with dry hydrogen or an inert gas, and heated to the brazing temperature in a furnace. Figure 7 shows a typical charge emerging from a furnace.²

²At Solar Aircraft Co., San Diego, Calif., where most of the furnace brazing was done.

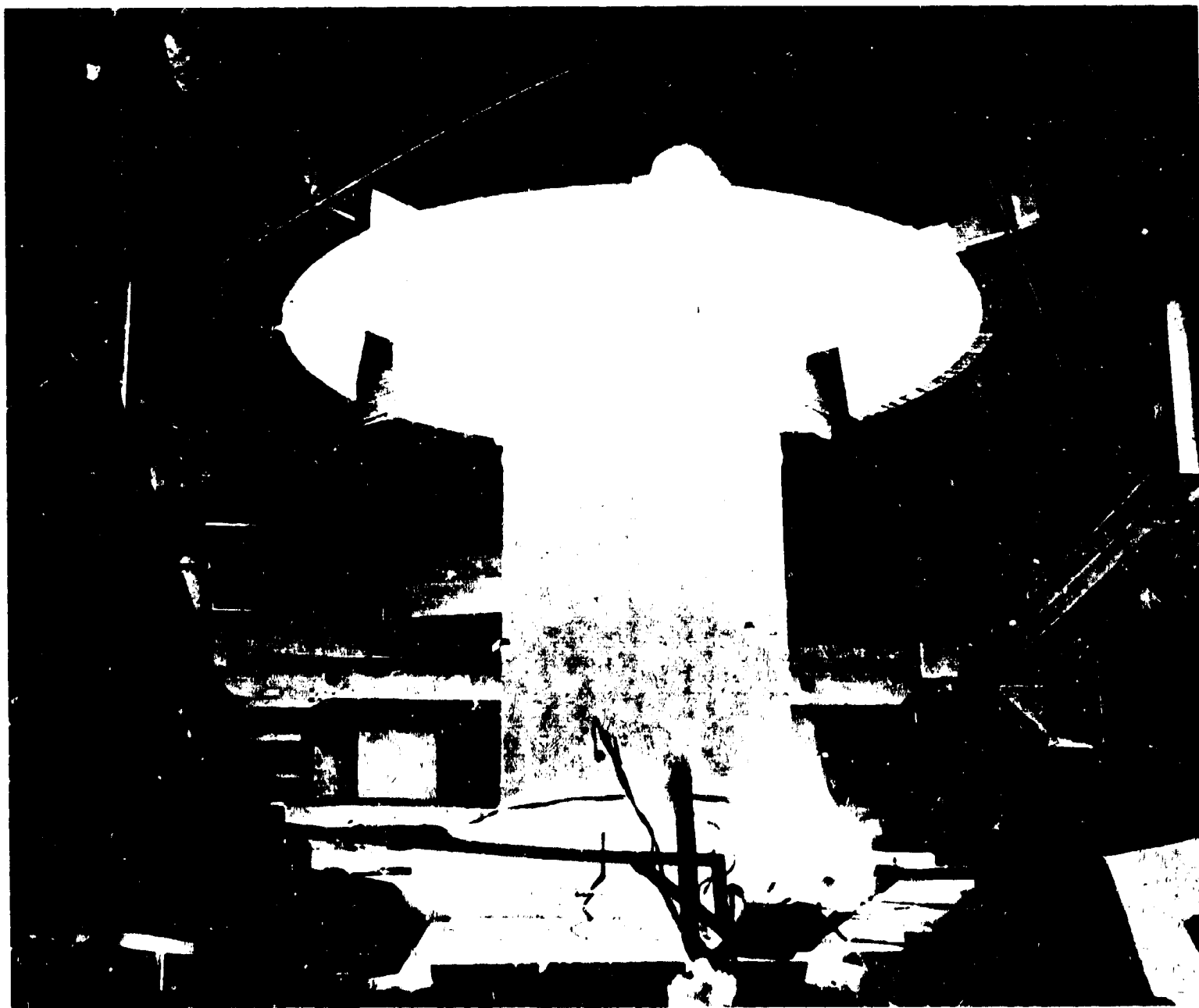


Fig. 7. Muffle emerging from brazing furnace.

2. **Mandrels.** The brazing mandrel must be carefully designed and fabricated. The following criteria apply:

1. The mass of the mandrel should be minimized and properly apportioned.
2. Dimensional stability and matched coefficients of expansion must be considered.
3. A continuous contact surface is preferable.
4. Over-all simplicity is desirable.

Ordinarily, mandrels are used only during the first braze cycle. If subsequent braze cycles are required, the tube bundle not only maintains its integrity but also retains its dimensions. This is to be expected, since furnace brazing heats the parts uniformly. In addition, most of the nickel-base brazing alloys have a high remelt temperature. The composition of the alloy changes during brazing due to diffusion, resulting in a remelt temperature that may be several hundred degrees Fahrenheit above the original value.

Ideally the mandrel should not be used even during the first braze cycle. However, for practical purposes it is usually required. If the tubes could be designed to be self-aligning and locking, and could then be intermittently tack-welded or banded together, the mandrel might be eliminated, except for the initial set-up.

3. **Brazing alloys.** According to Rose and Lewis (Ref. 2) an ideal brazing alloy has several attributes besides that of being capable of producing a strong joint. It should melt completely at a single temperature or within a narrow temperature range. When molten, it should wet and flow freely, but there should be a minimum of alloying between the base metal and the filler alloy. In addition, the Brazing Manual (Ref. 3) suggests that the filler metal have "... sufficient homogeneity and stability to minimize separation by liquidation ..." While none of the alloy systems discussed below are ideal, most of them have considerable practical use.

In the early stages of the 6K development program when torch brazing was contemplated, several silver-base and gold-base alloys were evaluated. Small test bundles for a 200-lb-thrust motor were brazed using representative alloys of both systems. However, the high flow temperature of the gold-base alloys made their use impractical.

For furnace brazing, the nickel-base alloys that qualified under AMS-4775, AMS-4776, AMS-4777, and AMS-

4778 were tested for compatibility with the hydrazine-nitrogen tetroxide system. Table 1 indicates the relative compatibility of various brazing filler metals with hydrazine. AISI 347 stainless steel was used as a comparison standard. The tests were run in a hydrazine bomb at 400°F. The compatibility factor R is defined as follows:

$$R = \frac{P}{At}$$

where

P = bomb pressure, psig

A = area of the bomb plus sample, in.²

t = time in minutes to arrive at 500 psig

The nickel-base alloys were developed primarily for elevated-temperature service. In a regeneratively-cooled

Table 1. Results of hydrazine compatibility tests

Material	Composition	Compatibility Factor R^a
Alloys		
AISI 347	18Cr-10Ni-Cb ^b , Bal Fe	2.3
AISI 4130	0.30C-0.50Mn-0.90Cr-0.20Mo, Bal Fe	10.0
AISI 4130, Rusty	0.30C-0.50Mn-0.90Cr-0.20Mo, Bal Fe	28.5
AISI 1020, Rusty	0.20C-0.90Mn, Bal Fe	16.0
AISI 1020, w/ Electroless Ni	0.20C-9.90Mn, Bal Fe	1.5
Molybdenum	Commercially pure molybdenum	1.0
2014-T3	4.5Cu-0.8Si-0.8Mn-0.4 Mg, Bal Al	1.8
Brazing Filler Metals		
Easy-Flo	18Cd-15Cu-16.5Zn-50Ag	1.5
AMS-4775	4Si-16.5Cr-4Fe-3.8B, Bal Ni	2.4
AMS-4776	4Si-16.5Cr-4Fe-3.8B, Low C, Bal Ni	3.7
AMS-4777	4Si-7Cr-3Fe-3B, Bal Ni	3.7
AMS-4778	4Si-2.6B, Bal Ni	4.1
NX-1	Nickel-base alloys ^c	0.5 ^d
RX-1		0.4 ^d
R-1		0.5 ^d
R-3		0.5 ^d
H		0.5 ^d

^aThe compatibility factor R is the reaction rate of the material with hydrazine at 400°F. in psi/m²/min. See text for detailed explanation.

^bPercentage composition of columbium is at least ten times that of carbon in the alloy.

^cProprietary alloys of Solar Aircraft Co., San Diego, Calif.

^dThese low values indicate an inhibiting effect of the filler metal on the decomposition rate of hydrazine.

motor, this property is ordinarily not required. The tube-to-tube joints function more as seals than as structural joints. If the joints are more than about 0.003 in. wide, brittle secondary phases may form.

The true brittleness of some nickel-base brazed joints is not apparent in the assembled chamber. This phenomenon is due primarily to the great ductility of the austenitic stainless steel tubes. The joints are small compared to the tubes and other components. Figure 8a

shows a cross-sectional view of such a joint. The transgranular crack follows the secondary phase and terminates in the more ductile matrix. Under the loading conditions experienced by the motor during proof testing and firing, these cracks do not seem to propagate.

Also, alloys such as AMS-4778 can severely erode thin-wall tubes when the braze alloy is grossly applied or the run-off collects. Figure 8b is a photomicrograph of thin-wall AISI 347 tubes brazed with AMS-4778 for 25 minutes



Fig. 8. Photomicrographs of AISI 347 brazed tubes in cross section showing brazing defects: a. Trans-granular crack in secondary phase of filler metal; b. Gross solutioning effect of filler metal.

at 1975°F in a hydrogen atmosphere. This is a cross-section through the sample end, where the braze alloy accumulated. Approximately 50% of the tube wall has been dissolved. Excessive use of braze alloy should be avoided. If more than one braze cycle is required, the part should be loaded lightly for the first cycle; the pinholes in the joint can be repaired during the next cycle.

Under certain conditions of temperature and braze-alloy application, there is a tendency to remelt, flow sluggishly, and solidify in "blobs." Figure 9 illustrates a case in which these protuberances were ground off flush with the tubes. This condition can be alleviated by minimizing braze-alloy application, by careful temperature-time control, and by proper atmosphere regulation.

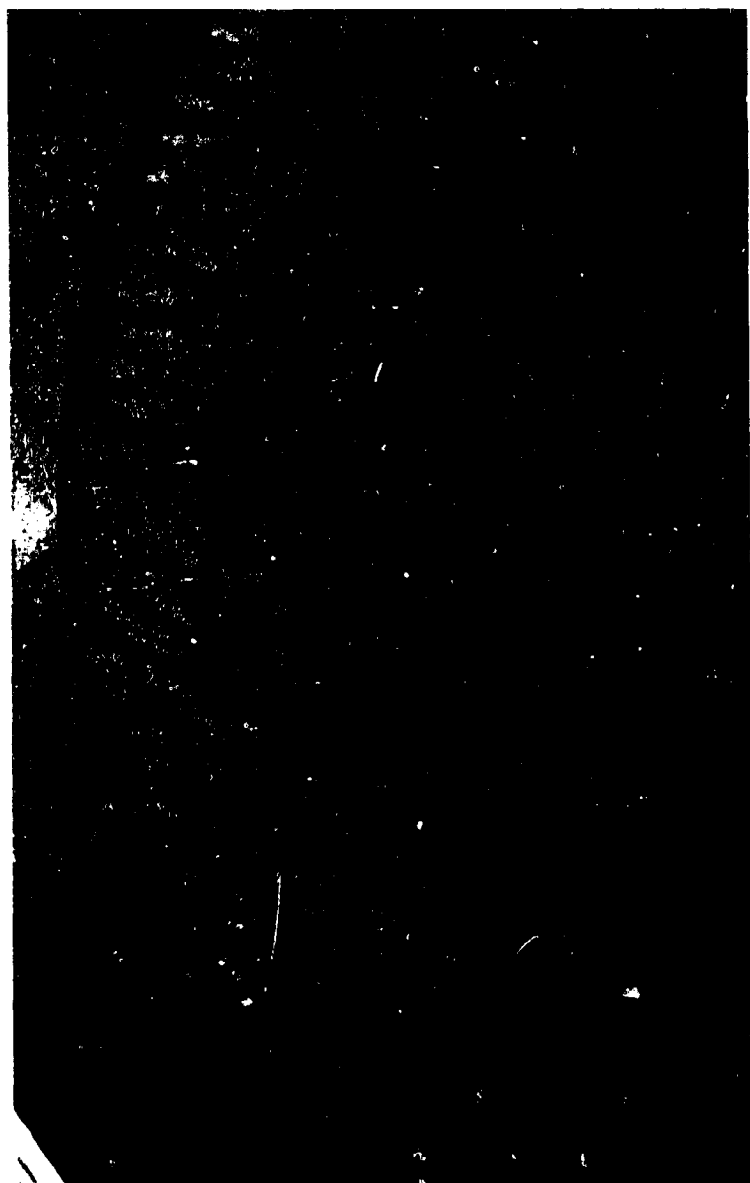


Fig. 9. Build-up of filler metal in brazed chamber (6K-J motor).

D. Incidental Fabrication Problems

1. **Exit skirt.** An exit skirt made from A-nickel is brazed into the 6K chamber to maintain the exit diameter accurately and to act as a flame shield. Figure 10 shows a 4:1 chamber with a scalloped skirt. It was very difficult to obtain a good fit between the tube bundle and the skirt. The teeth of the skirt could be machined quite accurately but maintaining the correct orientation of the tubes was difficult. The slightest twist or longitudinal shifting made the assembly of the skirt after the first braze cycle almost impossible. The problem was temporarily solved by using a nickel powder filler with the braze alloy.

The final solution to the problem consisted of tapering the skirt to a knife edge and forming it into the exit cone of the partially brazed chamber. The skirt was then spotwelded to the tubes, replacing the retaining ring formerly used. This procedure is depicted in Fig. 11.

2. **Stiffener rings.** For structural reasons stiffener rings were required for the aft bell of the 6K 20:1-expansion motor. Figure 12 shows the first approach to the problem of assembling these rings for brazing. This was obviously unsatisfactory because the force applied to the outer flange pulled the rings away from the bundle. A second approach (Fig. 13) was spotwelding the tie-down

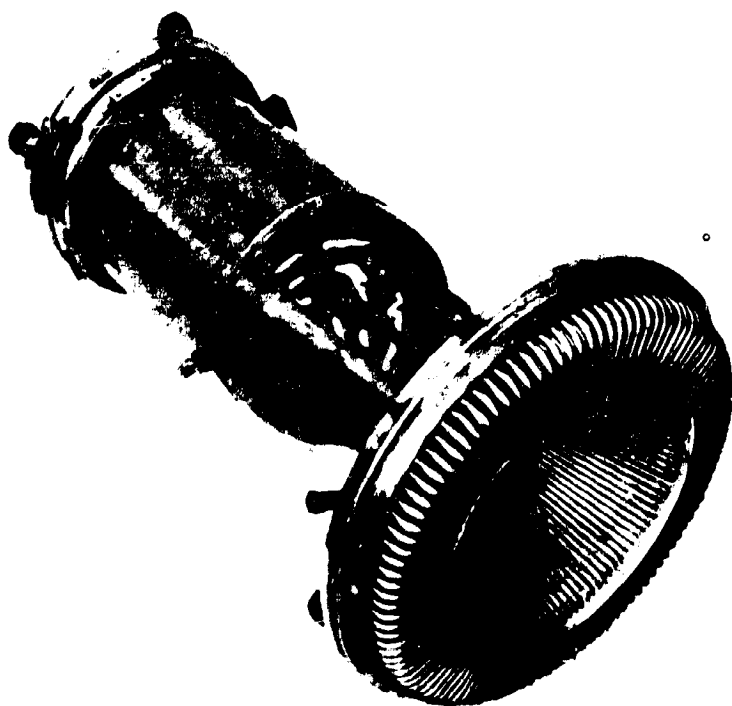


Fig. 10. Completed chamber showing aft skirt (6K-J).

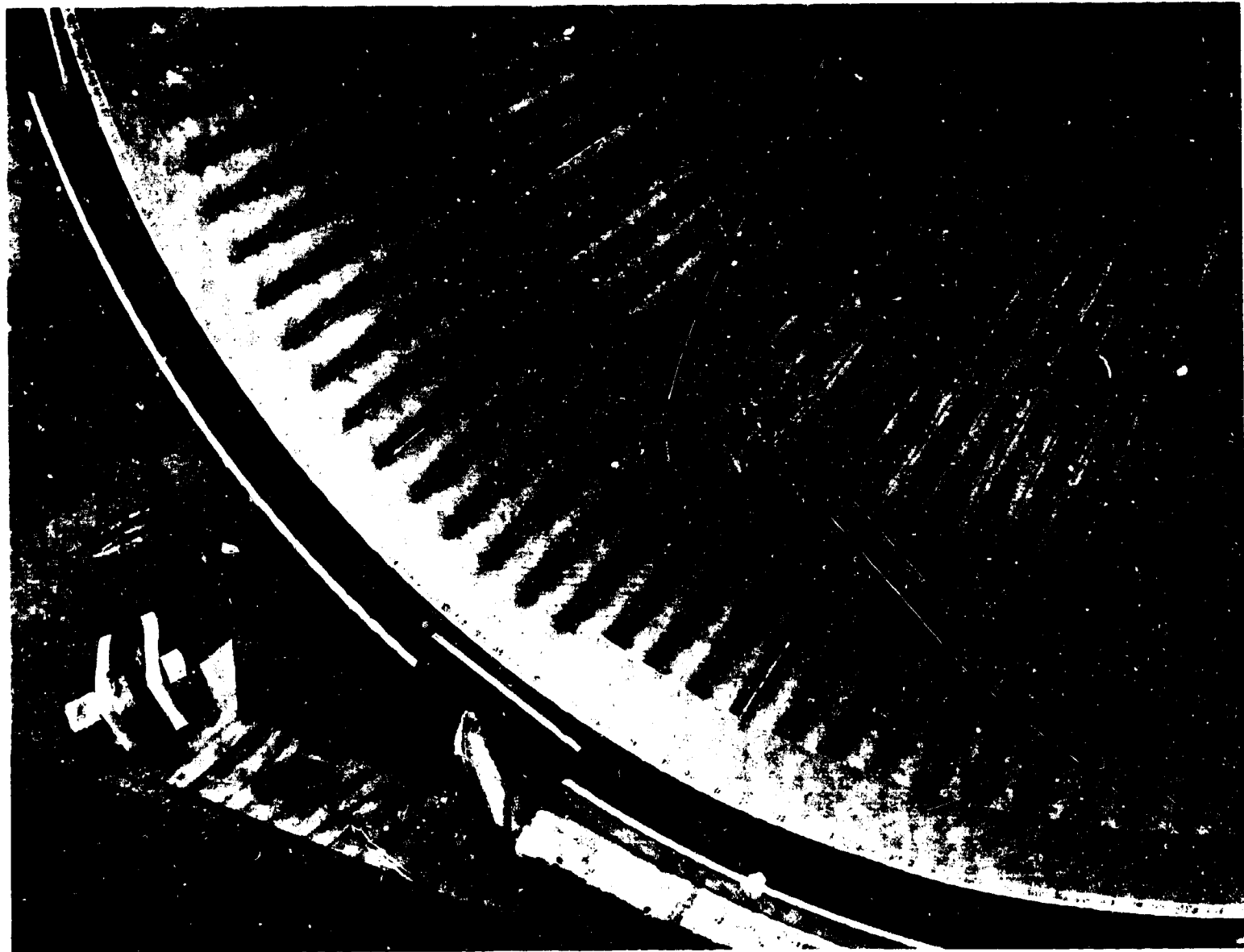


Fig. 11. Tapered aft skirt (6K-H motor).

wire to the conical flange. However, there was still a noticeable tendency towards pulling away at the six points where the load was applied. A third attempt (Fig. 14) utilized tooling rings. The load was applied evenly to the stiffener rings but the tooling rings deflected. This in turn was corrected by redesigning the rings as illustrated in Fig. 15.

Another procedure consisted of spotwelding a wire or band to the tubes just above the stiffener rings to prevent them from shifting during brazing. The tooling rings can then be removed and the unit brazed. The elimination of the heavy tooling rings resulted in greater temperature uniformity.

3. **Wire-wrapping.** The chamber section of the motor is solidly wound with Haynes Alloy No. 25 wire while

the aft bell is intermittently wound with AISI 302 stainless steel. The latter is spotwelded and brazed to the tubes but the former is attached only at small turnpost. The chamber-section wire carries hoop loads while the aft bell wire (1) carries hoop loads, (2) prevents the bell section from buckling where the tension loads change to compression loads, and (3) prevents the "kidney" cross-section of the tube from deforming when pressurized.

Figure 16 shows the wire being spotwelded to the aft bell of the 6K 20:1-expansion motor after the first braze cycle. At each point where the wire contacts a tube, braze alloy was placed. About 30,000 spots were applied to each motor. Several other methods of carrying the hoop loads were considered, including glass-filament winding

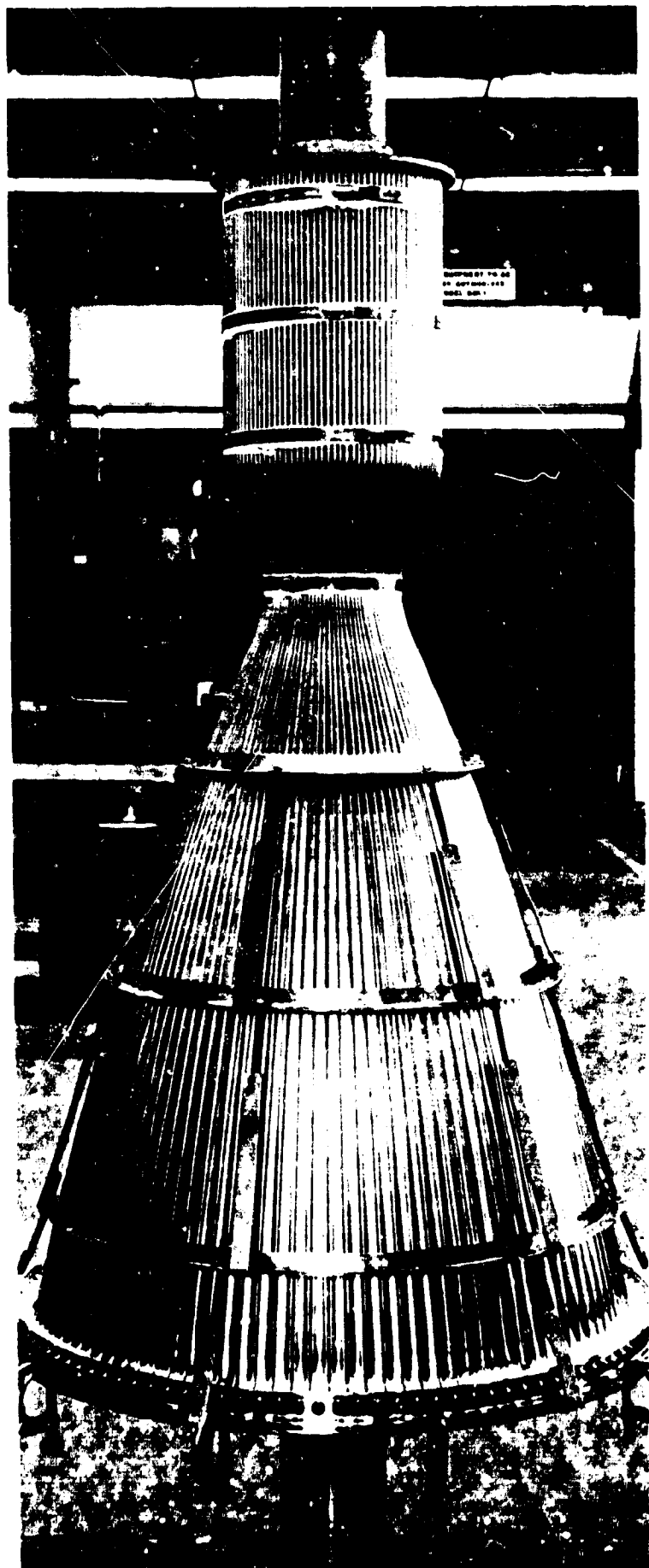


Fig. 12. Assembly of stiffener rings, first method.

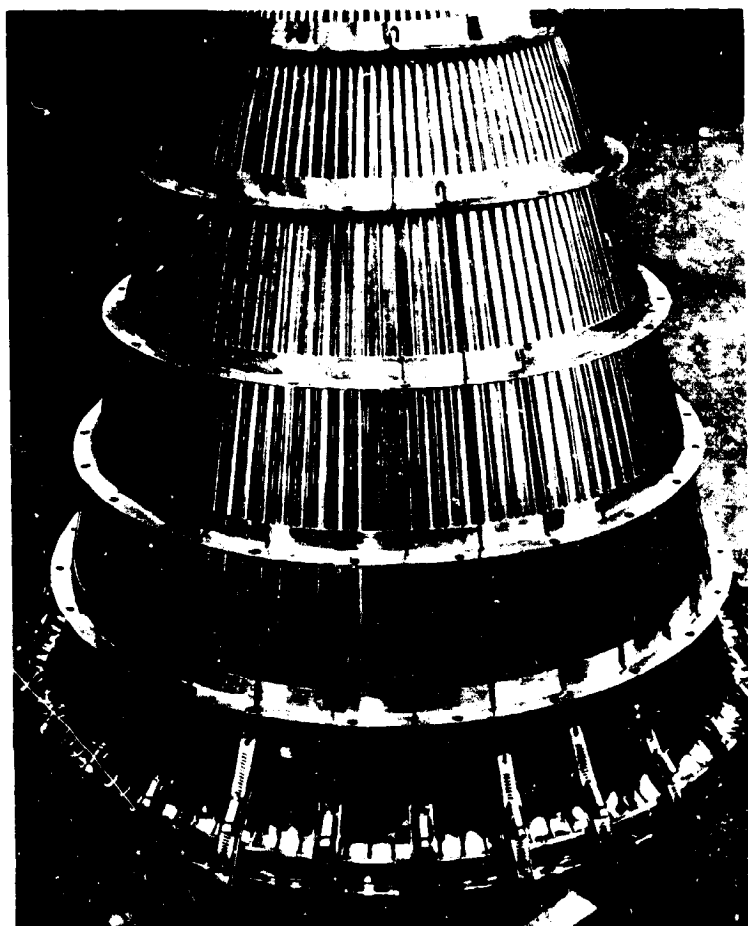


Fig. 13. Assembly of stiffener rings, second method.

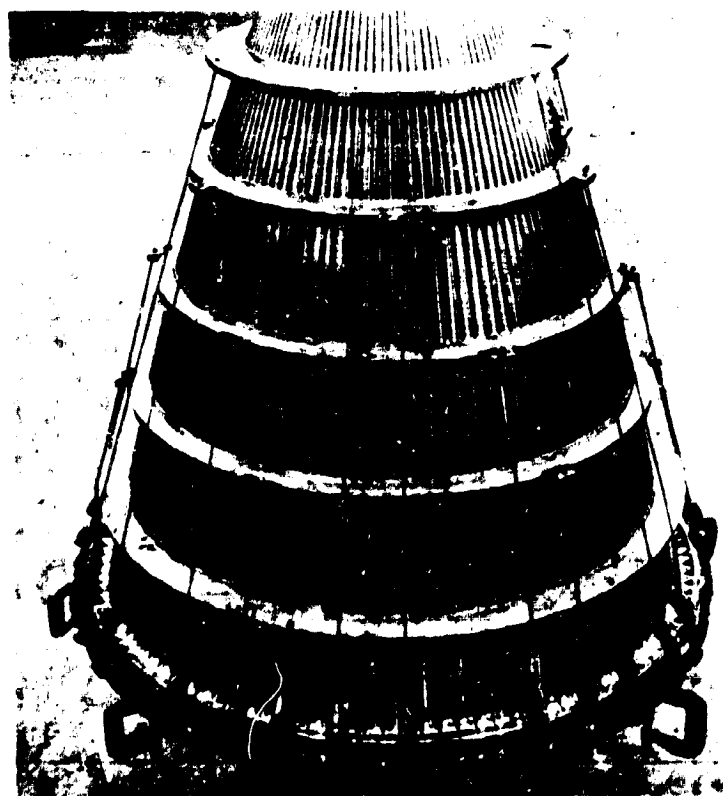


Fig. 14. Assembly of stiffener rings, third method.

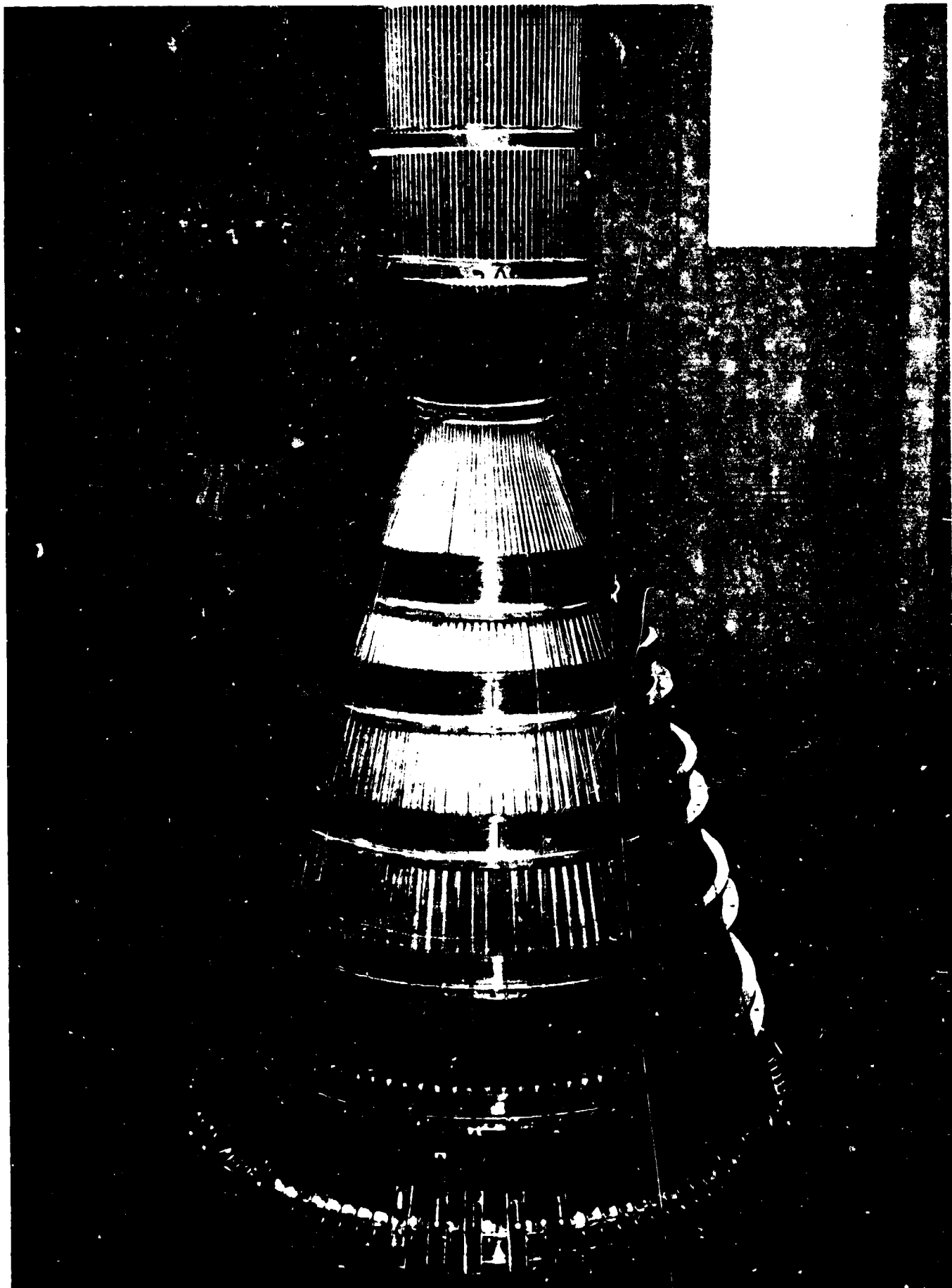


Fig. 15. Ultimate method of applying pressure to stiffener rings for assembly.

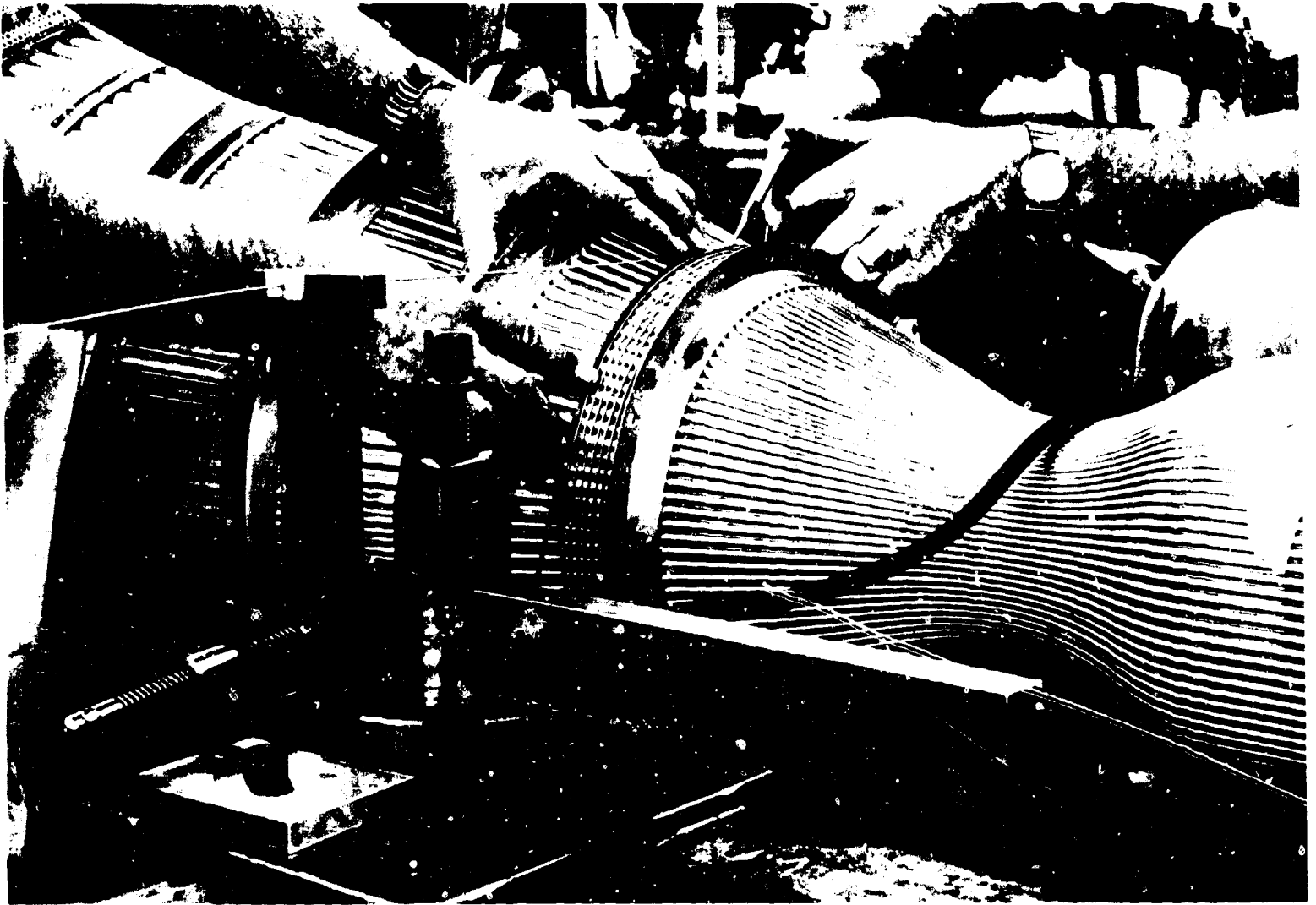


Fig. 16. Spotwelding wire wrapping to aft bell of 20:1-expansion chamber.

with epoxy binder, glass-tape winding with epoxy binder, and thin perforated-metal-tape wrapping.

4. **Chamber coating.** The chamber is coated with a polysulfide-modified epoxy to protect the wire, to act as a moisture barrier, and to prevent wire crossing under load. The coating is applied by brush while the chamber is rotated on a turn-table. It is cured at room temperature.

The polysulfide elastomer provides sufficient ductibility to prevent cracking under normal operating conditions.

Prior to the selection of the modified epoxy, attempts were made to coat the wire by silver brazing and with an unmodified epoxy. Neither selection was satisfactory. The coating presently used is not perfect, but it is satisfactory enough not to warrant change during the development stage of the program.

III. FUTURE DEVELOPMENT

There are many aspects of the 6K system amenable to future refinement or development. Two of these will be discussed briefly. The first goes back to the beginning of the fabrication process, to tube development; the second, at the other extreme, deals with materials and processes involved in the development of an uncooled nozzle extension.

A. Tubes

The stainless steel tubes are the basic skeleton of the thrust chamber. Their refinement is therefore vital to the general improvement of the engine. The tubes have evolved along two principal lines:

1. The quality of the tubes has been improved by electrolytic grinding, eddy-current inspection, better forming techniques, etc.
2. The configuration of the tubes has been altered several times to improve the performance of the system.

In the future the following items will be investigated:

1. Alloys with high strength-to-weight ratio.
2. Vacuum-melted alloys for higher-quality tubing.
3. Aluminum alloys.
4. The interaction of heat-treatment and brazing cycles.

B. Nozzle Extensions

Most of the problems associated with the fabrication and sizing of the 4:1-expansion tubes have been solved. The complex drawing, contouring, and sizing of the 20:1-expansion tubes require somewhat greater effort. To supplement this approach, uncooled extension nozzles are being considered.

Ablative materials and refractory metals are very promising. Figure 17 shows an 8:1 extension nozzle bolted to a 6K 4:1-expansion motor. This nozzle is constructed from a Refrasil reinforced epoxy liner and a standard fiberglass reinforced epoxy outer shell. To date, the test-firing results have been encouraging. Refractory metals which look promising include molybdenum, niobium, tantalum, and possible tungsten.

A full 20:1-expansion skirt fabricated from molybdenum is planned for the near future. The following problems are anticipated:

1. Lack of room-temperature ductility necessitates strict handling precautions.
2. Good oxidation-resistant coatings for extended operation are not fully developed.
3. The attachment problem may require complex joint design.
4. The state of the art does not permit reliable welded assemblies to be satisfactorily coated.
5. Material costs are high.
6. The lightweight structure may be subject to resonant dynamic loads.

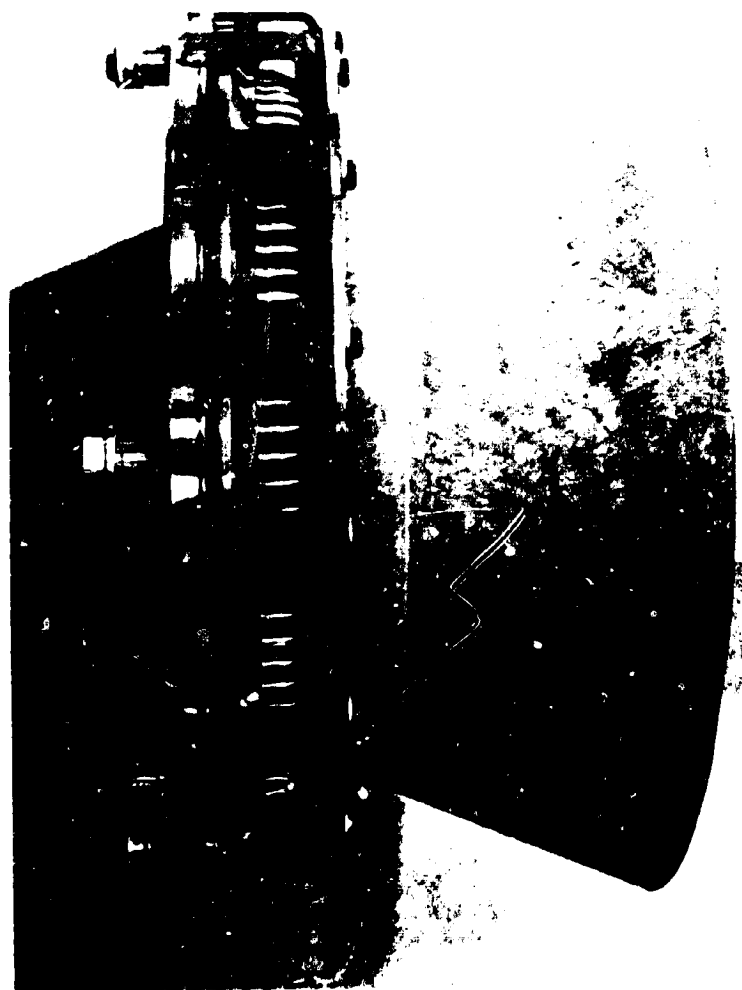


Fig. 17. Reinforced-epoxy nozzle extension on 4:1-expansion chamber.

IV. CONCLUSION

The success of a development program depends—among other things—upon attention to detail, control of variables, recognition of the true program goals, and the proper balance between conservative engineering and daring foresight. A detailed analysis of the interplay of variables is important to the swift determination and correction of troubles. Unless the available manpower permits, the system should not be optimized until the basic concepts have been developed.

Briefly, the materials problems associated with the development of a tube-bundle motor, from the evolution of the stainless steel tubes to the furnace brazing of those tubes into a motor, have been reviewed. Ancillary problems, concerning the exit skirt, stiffener rings, wire wrapping, and coating, were discussed. Even more challenging problems will undoubtedly be encountered in the refinement of this system for space-flight missions.

REFERENCES

1. Chang, W. H. "Basic Characteristics of Some Heat-Resistant Brazing Filler Metals." *Welding Journal*, Vol. 35, No. 9, September 1956.
2. Rose, A. S. and Lewis, W. N. "High Temperature Alloy Brazing of Thin Materials for Jet Engines." *Welding Journal*, Vol. 34, No. 1, January 1955.
3. *Brazing Manual*, American Welding Society. Reinhold Publishing Corporation 1955.
4. Aggen, G. et al., "Ni-Cr-B Brazing of a High-Temperature Alloy." *Welding Journal*, Vol. 36, No. 8, August 1957.